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ABSTRACT (Maximum 200 words)

This report summarizes the research and development that has been carried out at Lawrence Berkeley Laboratory on metal ion implantation for a number of applications. The technical objectives of the program were to investigate the surface modification of metals, ceramics, and other materials by energetic implantation of metal ions and by the deposition of metal plasma, and the interaction of these two processes when applied sequentially or simultaneously. Metal ion implantation was done with a vacuum arc ion source based implantation facility that was developed under prior ARO/ONR funding, and the metal plasma deposition was done using vacuum arc plasma guns. Gaseous species could be added during the implantation and deposition processes as a means of incorporating non-metallic elements into the surface and thus forming films and modified zones (implanted surfaces layers) of compound materials such as oxides and nitrides. Several novel research topics were addressed, as well as one specific sizeable project involving the surface modification of long copper rails from an electromagnetic rail gun.

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SURFACE MODIFICATION
BY HIGH CURRENT METAL ION IMPLANTATION

Final Report

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Ian G. Brown

March, 1995

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The view, opinions, and/or findings contained in this report are those of the author and should not be construed as an official Department of the Army position, policy, or decision, unless so designated by other documentation.

A. STATEMENT OF THE PROBLEM STUDIED

The technical objectives of this program were to investigate the surface modification of metals, ceramics, and other materials by energetic implantation of metal ions and by the deposition of metal plasma, and the interaction of these two processes when applied sequentially or simultaneously. Metal ion implantation was done with a vacuum arc ion source based implantation facility that was developed under prior ARO/ONR funding, and the metal plasma deposition was done using vacuum arc plasma guns. Gaseous species could be added during the implantation and deposition processes as a means of incorporating non-metallic elements into the surface and thus forming films and modified zones (implanted surface layers) of compound materials such as oxides and nitrides. Several novel research topics were addressed, as well as one specific sizeable project involving the surface modification of long rails from an electromagnetic rail gun.

B. SUMMARY OF RESULTS

The work done can be divided into three primary parts:

- A number of independent, smaller research topics were addressed throughout the course of the three year program;
 - A specific major undertaking was the surface modification, in several stages of scale-up, of a number of relatively large (up to 5 feet long) copper rails from an Army electromagnetic rail gun facility;
 - Technology developments, involving the invention of new kinds of hardware.
- These three domains of work are addressed in the following.

Research topics

We studied the effect of titanium ion implantation on the pitting corrosion behavior of aluminum. The surface morphology of implanted samples was investigated with scanning electron microscopy, and the surface composition, oxide thickness and structure, and implanted titanium depth profiles were determined by Auger microscopy, XPS and RBS. The Ti-implanted aluminum showed an increased resistance to pitting corrosion over a range of implantation dose and energy. This work was presented at the 8th International Conference on Ion Beam Modification of Materials, Heidelberg, Germany, September 7-11, 1992, and was published in the proceedings.

Collaboratively with Dr. Eal Lee and coworkers at the Oak Ridge National Laboratory, we investigated the effect of metal ion implantation into polypropylene for modification of the surface wear properties. Our vacuum arc metal ion source provides a very nice tool for this application because of the ease with which high dose metal ion implants can be carried out. Here we used Ti and Ag at 100 keV. We found that the wear resistance could be significantly improved. A paper describing the work has been accepted for publication in J. Mater. Res.

Rare earth doping of semiconductors is important for the study and fabrication of optoelectronic materials. Here, too, our metal ion implantation facility offers a powerful tool. Collaboratively with optoelectronic researchers at Ohio University we've doped a wide range of substrates with a wide range of rare-earths. For example we've implanted species including Sc, Y, La, Ce, Pr, Nd, Sm, Gd, Dy, Ho, Er and Yb into host materials including Si, GaAs, InP and more. The implantation dose was varied from a low of about 10^{13} cm⁻² up to as high as about

10^{17} cm^{-2} , and the ion energy up to about 200 keV. A number of fundamentally important research investigations were made and reported on.

We have explored the effect of metal ion implantation into high speed steel pistons using a testing facility that simulated the wear-strain of the tools used in the metal forming industry. Hafnium was found to offer some advantages, but not by a large factor; maximum improvement was less than a factor of two.

Electromagnetic rail gun materials development

In a collaboration with Dr. Monde Otooni of the U.S. Army ARDEC at Picatinny Arsenal, N.J., (collaboration suggested and initiated by Dr. R. Reeber of ARO), we carried out a substantial program of investigation of the application and suitability of our metal ion implantation techniques for the improvement in the erosion characteristics of electromagnetic rail gun components. The rails and armatures of these devices are subject to severe erosion by electrical arcing and frictional wear. In the first phase of the investigation we implanted small samples of Cu and Al with various metal ion species and characterized the improvements in arcing and wear resistance using a simple spark testing apparatus and a scratch tester, both made specially for the purpose. The results were moderately encouraging, and the decision was made to proceed on to the next phase, involving the implantation of four 24-inch long copper rails, as well as a number of smaller aluminum armatures. The rail implantation project was a sizeable job. To accommodate the large pieces we set up our "very large area implantation facility", previously demonstrated but not used for an actual implantation. This involved installation of 50-cm diameter extractor grids for the formation of ion beams of about 1000 sq. cm. in area, at energy about 100 keV or more, and with peak beam current of several amperes, as well as modification to the vacuum vessel in order to be able to accommodate and manipulate the rail during the implantation process. Various metallic species (Ta, Cr, Ni and others) were implanted into several 24-inch rails. The implanted rails were tested in actual gun firings at Picatinny, and the arc erosion and wear damage were indeed reduced. Two papers reporting on the work were presented at an MRS conference (see list of papers). This project provided a very nice demonstration of the very large area vacuum arc ion source implantation technology developed by us.

The next major phase of this project involved the surface modification of yet larger rail components – 5 feet long. The size of the pieces involved puts this work at the forefront of world ion beam surface modification r&d. The 6 rails were each 5 ft long by 1 inch wide, for a total surface area of about 2500 sq. cm. The processing required was an implantation of tantalum into the copper at a very high concentration (several tens of percent) and over a deep range (up to about 1000 Å). It turns out that such an implantation is, in fact, simply not possible, due to sputtering of already-implanted material by the ion beam itself – the sputter-limited retained dose for Ta into Cu at energies in the hundred-to-several-hundred keV range corresponds to a maximum concentration of less than 5%, and the ion range at these (practical) implantation energies is less than 200 or 300 Å. To circumvent this inherent limitation on the kinds of surface modification that can be achieved by ion implantation, we developed and used a new technique which we've called 'pseudo-implantation'. The technique involves a metal-plasma-immersion-based method for synthesizing non-equilibrium alloy layers of arbitrarily high dopant concentration and of arbitrary thickness. By repetitively pulse biasing the substrate to high negative voltage while it is immersed in the metal plasma from a vacuum arc plasma gun, a layer can be synthesized that is atomically mixed into the substrate with an interface width determined by the early-time bias voltage and with a thickness determined by the overall duration of the process. The species is that of the vacuum arc cathode material, which for this purpose can be a mixture of the substrate metal and the wanted dopant metal, 50:50 Ta:Cu. We used a scaled-up vacuum arc plasma gun with a magnetic macroparticle filter to remove all droplets from the depositing plasma flux. The Ta depth profile was flat at

about 50at% Ta in Cu to a depth of about 1000 Å. This 'pseudo-implantation' technique offers a powerful method for the plasma synthesis of surface modified layers that are otherwise impossible to obtain, and it can be scaled up to large size as might be required for large-scale industrial application. The rail gun processing work done provided an excellent demonstration of the method. The rails are to be tested at Picatinny Arsenal.

Hardware developments

- The 50-cm diameter ion source and ion implanter embodiment was established as a useable, "real" implanter via its use for the rail gun implantation project described above. This 1000-cm² area ion beam facility was reported on in the literature. This configuration of implanter used part of the test facility with which we had previously demonstrated the viability of a high current, very broad beam, dc implanter, and which won us our second R&D-100 Award.
- We developed hardware to do combined metal plasma deposition and metal ion implantation simultaneously. A special embodiment of miniature low energy vacuum arc plasma gun was made in such a way that it could be located within the main vacuum vessel so as to deposit its metal plasma flux on the same target as to be implanted. At the same time, the plasma gun can be removed through a load-lock without exposing the sample to the atmosphere.
- A semi-automated set-up was developed for doing the "pseudo-implantation" processing (metal plasma immersion ion implantation and deposition) of the 6-foot long rails. A vacuum arc plasma gun was coupled to a magnetic filter ('plasma duct') to remove neutrals and macroparticles from the plasma stream, while a negative high voltage pulse bias was applied to the substrate during the deposition process so as to generate atomic mixing at the interface. The rail substrate was located 12 cm away from the duct exit, and was slowly moved lengthwise across the plasma stream so as to process the entire rail length.

C. PUBLICATIONS

Papers published in refereed journals, or presented at scientific conferences and subsequently published in refereed journals as part of the conference proceedings:

"Low Energy Metal Ion Source",
Xavier Godechot and Ian Brown,
J. Vac. Sci. Tech. A10, 254 (1992).

"Development of DC Broad Beam Mevva Ion Source",
I.G. Brown, M.R. Dickinson, J.E. Galvin and R.A. MacGill,
Rev. Sci. Instrum. 63, 2417 (1992).

"Metal Vapor Vacuum Arc Ion Sources",
Ian G. Brown, Rev. Sci. Instrum. 63, 2351 (1992);

"Anomalous Ranges of Ions Implanted into Carbon",
G. Dearnaley, J.L. Ing, S. Sugden, I.G. Brown and X. Godechot,
Surface and Coatings Technol. 51, 364 (1992).

"Distribution of Ion-Implanted Yttrium in Cr₂O₃ Scales and in the Underlying Ni-24wt%Cr",
Peggy Hou, Victor Chia and Ian Brown,
Surface and Coatings Technol. 51, 73 (1992).

"Versatile High Current Metal Ion Implantation Facility",
I.G. Brown, M.R. Dickinson, J.E. Galvin, X. Godechot and R.A. MacGill,
Surface and Coatings Technol. 51, 529 (1992).

"Plasma Synthesis of Metallic and Composite Thin Films with Atomically Mixed Substrate Bonding"
I.G. Brown, A. Anders, S. Anders, M.R. Dickinson, I.C. Ivanov, M.A. MacGill, X. Yao, and K.M. Yu,
Nucl. Instrum. Meth. Phys. Res. B80/81, 1281 (1993);
Invited Paper, 8th International Conference on Ion Beam Modification of Materials,
Heidelberg, Germany, September 7-11, 1992.

"The Pitting Corrosion Behavior of Aluminum Ion Implanted with Titanium"
X. Y. Yao, C. S. Kumai, T. M. Devine, P. B. Fojas, I. C. Ivanov, K. -M. Yu
and I. G. Brown
Nucl. Instrum. Methods B80/81, 267 (1993);
Presented at the 8th International Conference on Ion Beam Modification of Materials,
Heidelberg, Germany, September 7-11, 1992.

"Improvement in Wear Characteristics of Steel Tools by Metal Ion Implantation",
D.M. Rück, D. Boos and I.G. Brown,
Nucl. Instrum. Meth. Phys. Res. B80/81, 233 (1993);
Presented at the 8th International Conference on Ion Beam Modification of Materials,
Heidelberg, Germany, September 7-11, 1992.

"Vacuum Arc Ion Sources for Particle Accelerators and Ion Implantation",
Ian G. Brown,
IEEE Trans. Plasma Sci. 21, 537 (1993);
Invited Paper, 15th International Symposium on Discharges and Electrical Insulation in Vacuum,
Darmstadt, Germany, September 6-10, 1992.

"Development of a Very Large Scale Metal Ion Implantation Facility",
Ian G. Brown
Invited Paper presented at the 1992 Coatings for Advanced Heat Engines Workshop,
Monterey, CA, August 2-7, 1992.

"Metal Ion Implantation for Large Scale Surface Modification",
Ian G. Brown,
J. Vac. Sci. Tech. A11, 1480 (1993);
Invited Paper; 39th National American Vacuum Society Symposium,
Chicago, Illinois, November 9-13, 1992.

"Plasma Synthesis of Thin Films and Multilayers with Tailored Atomic Mixing",
Ian Brown in *Plasma Synthesis and Processing of Materials*, edited by K. Upadhy (pub. TMS,
Warrendale, PA, 1993), p. 37;
Invited Paper, TMS Conference on Plasma Synthesis and Processing of Materials,
Denver, Colorado, February 21-25, 1993.

"Tantalum Ion Implantation into Cu-12Nb for Electromagnetic Rail Gun Technology",
M.A. Otooni, S. Foner and I.G. Brown,
Mat. Res. Soc. Symp. Proc. 316, 569 (1994);
Materials Research Society Fall Meeting, Boston, MA, Nov 29 - Dec 3, 1993.

"Surface Modification of Electromagnetic Rail Gun Components",
M. A. Otooni, A. Graf, C. Dunham and I. G. Brown,
Mat. Res. Soc. Symp. Proc. 316, 585 (1994);
Materials Research Society Fall Meeting, Boston, MA, Nov 29 - Dec 3, 1993.

"Low Energy Ion Implantation / Deposition as a Film Synthesis and Bonding Tool",
A. Anders, S. Anders, I.G. Brown and I.C. Ivanov,
Mat. Res. Soc. Symp. Proc. 316, 833 (1994);
Invited Paper, Materials Research Society Fall Meeting, Boston, MA, Nov 29 - Dec 3, 1993.

"Poor Man's Scratch Tester",
R.A. MacGill, X.Y. Yao, R.A. Castro, M.R. Dickinson and I.G. Brown,
J. Vac. Sci. Tech. A11, 2856 (1993).

"Ion Induced Damage and Annealing of Single Crystal Zirconia Implanted with Zirconium, Yttrium
or Hafnium Ions",
D.X. Cao, D.K. Sood and I.G. Brown,
Mat. Res. Soc. Symp. Proc. 316, (1994);
Materials Research Society Fall Meeting, Boston, MA, Nov 29 - Dec 3, 1993.

"Metal Ion Implantation - Conventional vs. Immersion",
I. Brown, A. Anders, S. Anders, M. R. Dickinson and R. A. MacGill,
J. Vac. Sci. Tech. B12, 823 (1994);
First International Workshop on Plasma-Based Ion Implantation,
Madison, Wisconsin, August 4-6, 1993.

"Metal Plasma Immersion Ion Implantation using Vacuum Arc Plasma Sources",
A. Anders, S. Anders, I. G. Brown, M. R. Dickinson and R. A. MacGill,
J. Vac. Sci. Tech. B12, 815 (1994);
First International Workshop on Plasma-Based Ion Implantation,
Madison, Wisconsin, August 4-6, 1993.

"Doping of Rare-Earths by Ion Implantation and Related Techniques",
Ian G. Brown
Mat. Res. Soc. Symp. Proc. 301, 39 (1993);
Invited Paper, Materials Research Society Spring Meeting, San Francisco, CA, April 12-16, 1993.

"Kinetics and Quenching Mechanisms of Photoluminescence in Yb-doped InP"
H. J. Lozykowski, A. K. Alshawa and I. Brown
J. Appl. Phys. 76, 4836 (1994)

"Electroluminescence of Yb-Implanted InP"
A.K. Alshawa, H.J. Lozykowski and I. Brown
Materials Research Society Fall Meeting, Boston, MA, Nov 28 - Dec 2, 1994.

"Luminescence Properties of Yb-doped InP",
H. J. Lozykowski, A. K. Alshawa, G. Pomrenke and I. Brown,
Mat. Res. Soc. Symp. Proc. 301, 263 (1993);
Materials Research Society Spring Meeting, San Francisco, CA, April 12-16, 1993.

"Luminescence Properties of Yb- and Nd- Implanted CdS",
A. K. Alshawa, H. J. Lozykowski, T. Li and I. Brown,
Mat. Res. Soc. Symp. Proc. 301, 281 (1993);
Materials Research Society Spring Meeting, San Francisco, CA, April 12-16, 1993.

"Low Energy Vacuum Arc Ion Source",
I.G. Brown, S. Anders M.R. Dickinson, P.B. Fojas and R.A. MacGill,
Rev. Sci. Instrum. 65, 1260 (1994).

"Low Energy Ion Beam Extraction and Transport – Experiment-Computer Comparison",
P. Spädtke, I.G. Brown and P.B. Fojas,
Rev. Sci. Instrum. 65, 1441 (1994).

"Multiple-Species Ion Beams from Titanium-Hafnium Alloy Cathodes in Vacuum Arc Plasmas",
J. Sasaki, K. Sugiyama, X. Yao and I. Brown,
J. Appl. Phys. 73, 7184 (1993).

"Vacuum Arc Ion Sources – Some Vacuum Arc Basics and Recent Results",
S. Anders, A. Anders and I. G. Brown,
Rev. Sci. Instrum. 65, 1253 (1994);
Invited Paper, 5th International Conference on Ion Sources,
Beijing, China, Aug 31 - Sep 4, 1993.

"Vacuum Arc Ion Source with Filtered Plasma for Macroparticle-Free Implantation",
S. Anders, A. Anders, I. G. Brown, R. A. MacGill and M. R. Dickinson,
Rev. Sci. Instrum. 65, 1319 (1994);
5th International Conference on Ion Sources, Beijing, China, Aug 31 - Sep 4, 1993.

"Broad Beam Extraction from Vacuum Arc Ion Sources",
I. G. Brown, S. Anders, A. Anders, M. R. Dickinson, R. A. MacGill and X. Yao,
Workshop on Mevva Ion Sources and Applications, Beijing Normal University,
Beijing, China, Aug 28 - 30, 1993.

"Some Observations of Vacuum Arc Ion Source Suppressor Grid Characteristics",
I. G. Brown, P. B. Fojas and P. Spädtke,
Workshop on Mevva Ion Sources and Applications, Beijing Normal University,
Beijing, China, Aug 28 - 30, 1993.

"Triggering Unit for Pulsed Vacuum Arc Ion Source",
A. Anders, S. Anders, I. Brown, G. J. deVries, G. W. Leonard, T. A. McVeigh,
M. L. Rickard and X. Yao,
Workshop on Mevva Ion Sources and Applications, Beijing Normal University,
Beijing, China, Aug 28 - 30, 1993.

"Platinum Ion Implantation into Single Crystal Zirconia with Carbon Sacrificial Layer on the Surface"

D.X. Cao, J.W. Chu, A.P. Pogany, D.K. Sood and I.G. Brown
Materials Research Society Fall Meeting, Boston, MA, Nov 28 - Dec 2, 1994.

"Thermal Annealing of Single Crystal Zirconia Implanted with Platinum Ions",
D. X. Cao, D. K. Sood and I.G. Brown,
Nucl. Instrum. Method Phys. Res. B91, 280 (1994).

"Vacuum Arc Ion Sources",
I. G. Brown,
Invited Review Paper, Rev. Sci. Instrum. 10, 3061 (1994).

"Effects of Metal Ion Implantation on Wear Properties of Polypropylene",
G.R. Rao, E.H. Lee, X. Yao and I.G. Brown,
J. Mat. Res. (to be published, 1995).

"Ion Beam Mixing of Metal Thin Films with Energetic Metal Ions"
F. J. Paoloni, K. M. Yu, Z. Wang, I. G. Brown and P. J. Evans
9th International Conference on Ion Beam Modification of Materials
Canberra, Australia, Feb 5-10, 1995.

"Synthesis of Unattainable Ion Implantation Profiles — "Pseudo-Implantation""
I. G. Brown, A. Anders, S. Anders, R. A. Castro, M. R. Dickinson, R. A. MacGill and
Z. Wang,
9th International Conference on Ion Beam Modification of Materials
Canberra, Australia, Feb 5-10, 1995.

The following papers were presented at conferences and have been published in abstract form only:

"Metal Vapor Vacuum Arc Ion Source (Mevva) for Metal Ion Implantation",
B.H. Wolf, H. Ernig, D.M. Rück, P. Spädtke, W. Fischer, H. Wituschek and I.G. Brown,
8th International Conference on Ion Beam Modification of Materials,
Heidelberg, Germany, September 7-11, 1992.

"Very Broad Beam Metal Ion Source for Large Area Ion Implantation Application",
I. G. Brown, S. Anders, M. R. Dickinson, R. A. MacGill and X. Yao,
IEEE Conf. Proc. Cat. No. 93CH3334-0 (1993), p. 212 (abstract only);
20th IEEE International Conference on Plasma Science, Vancouver, Canada, June 7-9, 1993.

"High Charge State Metal Ion Production in Vacuum Arc Ion Sources",
I.G. Brown, A. Anders and S. Anders,
Invited Paper presented at the 21st IEEE International Conference on Plasma Science,
Santa Fe, NM, June 6-8, 1994.

"Metal Ion Implantation",
Ian Brown,
Invited Paper presented at the Workshop on Status and Future Developments of Ion Technologies"
Garmisch-Partenkirchen, Germany, September 19, 1994.

Book Chapters:

"Fundamental Processes" and "Ion Source Characterization - Vacuum Arc Ion Sources",
Ian Brown, in *Handbook of Ion Sources*, B. H. Wolf. ed., CRC Press, Boca Raton, Fla, 1995
(to be published).

"Pulsed Arc Sources" and "Pulsed Ion Sources",
Ian Brown, in *Vacuum Arc Science and Technology*,
R.L. Boxman, P. Martin and D. Sanders eds, Noyes, N.Y., 1995 (to be published).

"Particle Sources - Ion", Ian Brown, in *Encyclopedia of Applied Physics*, VCH/AIP Press,
N.Y., 1995 (to be published).

D. PARTICIPATING PERSONNEL

Participants in this research program were:

Ian Brown, (PI), senior physicist
André Anders, physicist
Simone Anders, physicist
Robert MacGill, senior technical associate
Michael Dickinson, technical associate

There were no postdoctoral researchers, graduate students, nor visiting researchers supported under this program. However, various guest scientists from a number of different laboratories and universities in the United States and overseas visited the laboratory and participated in various aspects of the research program for various lengths of times at their own expense (ie, costs paid by their own institution).

The following scientists visited my laboratory to participate in collaborative research as indicated:

- (i) Dr. Frank Paoloni, University of Wollongong, Australia, for a 2-month stay as a participating guest, to participate in research related to ion mixing.
- (ii) Dr. Peter Spädtke, Gesellschaft für Schwerionenforschung (GSI), Darmstadt, Germany, for a 1-week stay, to participate in experiments on the ion charge state distribution of the ion beam produced.
- (iii) Dr. Dorothee Rück, Gesellschaft für Schwerionenforschung (GSI), Darmstadt, Germany, for a 2-week stay, to participate in experiments on ion implantation into steel for surface hardening.
- (iv) Dr. Dinesh Sood, from the Royal Melbourne Institute of Technology, Melbourne, Australia, for several 1-week stays, to participate in experiments related to ion implantation into ceramics.
- (v) Dr. Dexin Cao, from the Royal Melbourne Institute of Technology, Melbourne, Australia, for a 3-month stay, to participate in experiments related to ion implantation into ceramics.

APPENDIX

OTHER ACTIVITIES

- Program Committee member, IEEE Particle Accelerator Conference, Dallas, TX, June '95.
- International Advisory Committee member, International Symposium on Beam Technologies, Dubna, Russia, March '95.
- International Advisory Committee member of the International Conference on Ion Sources (ICIS'95), Vancouver, Canada, September 10-16, 1995.
- Editorial Board member, *Plasma Sources: Sci. & Techol.*, 1994-96.
- Chairman, Symposium on Industrial Applications of Ion Beams, 1994 APS General Meeting, Washington, DC, March '94.
- IEEE Nuclear & Plasma Sciences Society; Plasma Science & Applications Executive Committee
 - Executive Committee Member, 1991-93
 - Deputy Chairman, 1993
- International Advisory Committee and Program Committee member, International Conference on Ion Sources, Beijing, August 1993.
- Elected to Scientific Membership of the Böhmische Physical Society "for original research in ion and plasma synthesis of high performance coatings"; 1993.
- Session organizer for Plasma, Ion and Electron Sources, 1993 IEEE International Conference on Plasma Science, Vancouver, Canada, June '93.
- Lecturer on Ion Sources, U.S. Particle Accelerator School, Austin, TX, January '92.
- Session organizer for Plasma, Ion and Electron Sources, 1992 IEEE International Conference on Plasma Science, Tampa, Florida, June '92.
- 1992 R&D-100 Award for 'DC Broad-Beam High-Current Metal Ion Source' as 'one of the 100 most technologically significant new products of the year'.